

Serial No.: 10/569,566

Case No.: 21461P

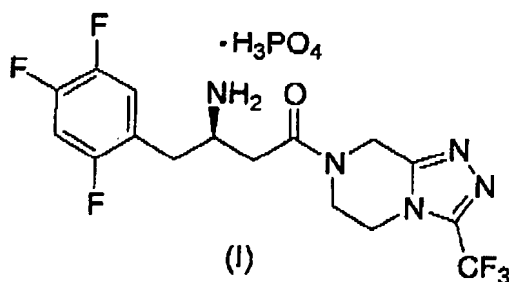
Page No.: 2

Amendments to the Claims:

This listing of claims will replace all prior versions, and listings, of claims in the application.

Listing of Claims:

Claim 1 (currently amended): A crystalline anhydrate form of the dihydrogenphosphate salt of (2*R*)-4-oxo-4-[3-(trifluoromethyl)-5,6-dihydro[1,2,4]triazolo[4,3-*a*]pyrazin-7(8*H*)-yl]-1-(2,4,5-trifluorophenyl)butan-2-amine of structural formula I:



~~characterized as being a crystalline anhydrate which is designated Form I characterized by characteristic reflections obtained from the X-ray powder diffraction pattern at spectral d-spacings of 18.42, 9.35, and 6.26 angstroms.~~

Claim 2 (cancelled)

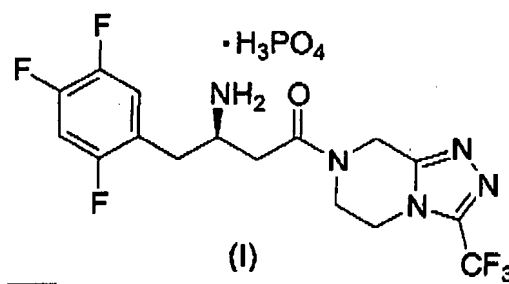
Claim 3 (currently amended): The crystalline anhydrate ~~Form I~~ of Claim 1 2 further characterized by characteristic reflections obtained from the X-ray powder diffraction pattern at spectral d-spacings of 5.78, 4.71, and 3.67 angstroms.

Claim 4 (currently amended): The crystalline anhydrate ~~Form I~~ of Claim 3 further characterized by characteristic reflections obtained from the X-ray powder diffraction pattern at spectral d-spacings of 3.99, 2.71, and 2.66 angstroms.

Claim 5 (currently amended): The crystalline anhydrate ~~Form I~~ of Claim 4 further characterized by the X-ray powder diffraction pattern of FIG. 1.

Serial No.: 10/569,566
 Case No.: 21461P
 Page No.: 3

Claim 6 (currently amended): ~~A~~ The crystalline anhydrate ~~F~~form I of ~~Claim 1~~ of the dihydrogenphosphate salt of (2R)-4-oxo-4-[3-(trifluoromethyl)-5,6-dihydro[1,2,4]triazolo-[4,3-a]pyrazin-7(8H)-yl]-1-(2,4,5-trifluorophenyl)butan-2-amine of structural formula I:



characterized by a solid-state fluorine-19 MAS nuclear magnetic resonance spectrum showing signals at -65.3, -105.1, and -120.4 p.p.m.

Claim 7 (currently amended): The crystalline anhydrate ~~F~~form I of Claim 6 further characterized by a solid-state fluorine-19 MAS nuclear magnetic resonance spectrum showing signals at -80.6, -93.5, and -133.3 p.p.m.

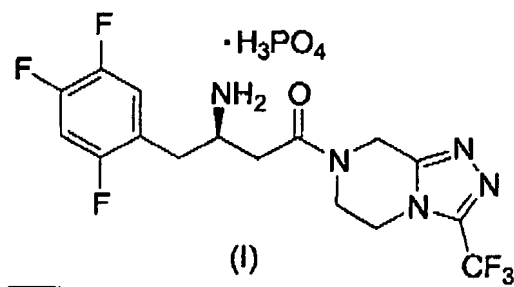
Claim 8 (currently amended): The crystalline anhydrate ~~F~~form I of Claim 7 further characterized by the solid-state fluorine-19 MAS nuclear magnetic resonance spectrum of FIG. 3.

Claim 9 (currently amended): ~~A~~ The crystalline anhydrate ~~F~~form I of ~~Claim 1~~ of the dihydrogenphosphate salt of (2R)-4-oxo-4-[3-(trifluoromethyl)-5,6-dihydro[1,2,4]triazolo-[4,3-a]pyrazin-7(8H)-yl]-1-(2,4,5-trifluorophenyl)butan-2-amine of structural formula I:

Serial No.: 10/569,566

Case No.: 21461P

Page No.: 4

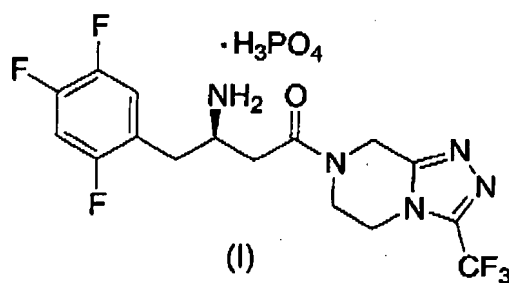


characterized by the solid-state carbon-13 CPMAS nuclear magnetic resonance spectrum of FIG. 2.

Claim 10 (currently amended) The crystalline anhydrate Form I of Claim 1 characterized by the thermogravimetric analysis curve of FIG. 5.

Claim 11 (cancelled)

Claim 12 (withdrawn) A dihydrogenphosphate salt of (2R)-4-oxo-4-[3-(trifluoromethyl)-5,6-dihydro[1,2,4]triazolo[4,3-a]pyrazin-7(8H)-yl]-1-(2,4,5-trifluorophenyl)butan-2-amine of structural formula I:



characterized as being a crystalline anhydrate Form III.

Claim 13 (withdrawn) The crystalline anhydrate Form III of Claim 12 characterized by characteristic reflections obtained from the X-ray powder diffraction pattern at spectral d-spacings of 17.88, 6.06, and 4.26 angstroms.

Serial No.: 10/569,566

Case No.: 21461P

Page No.: 5

Claim 14 (withdrawn) The crystalline anhydrate Form III of Claim 13 further characterized by characteristic reflections obtained from the X-ray powder diffraction pattern at spectral d-spacings of 9.06, 5.71, and 4.55 angstroms.

Claim 15 (withdrawn) The crystalline anhydrate Form III of Claim 14 further characterized by characteristic reflections obtained from the X-ray powder diffraction pattern at spectral d-spacings of 13.69, 6.50, and 3.04 angstroms.

Claim 16 (withdrawn) The crystalline anhydrate Form III of Claim 15 further characterized by the X-ray powder diffraction pattern of FIG. 11.

Claim 17 (withdrawn) The crystalline anhydrate Form III of Claim 12 characterized by a solid-state fluorine-19 MAS nuclear magnetic resonance spectrum showing signals at -63.0, -103.1, and -120.2 p.p.m.

Claim 18 (withdrawn) The crystalline anhydrate Form III of Claim 17 further characterized by a solid-state fluorine-19 MAS nuclear magnetic resonance spectrum showing signals at -95.3, -98.7, -135.2, and -144.0 p.p.m.

Claim 19 (withdrawn) The crystalline anhydrate Form III of Claim 18 further characterized by the solid-state fluorine-19 MAS nuclear magnetic resonance spectrum of FIG. 13.

Claim 20 (withdrawn) The crystalline anhydrate Form III of Claim 12 characterized by the solid-state carbon-13 CPMAS nuclear magnetic resonance spectrum of FIG. 12.

Claim 21 (withdrawn) The crystalline anhydrate Form III of Claim 12 characterized by the thermogravimetric analysis curve of FIG. 15.

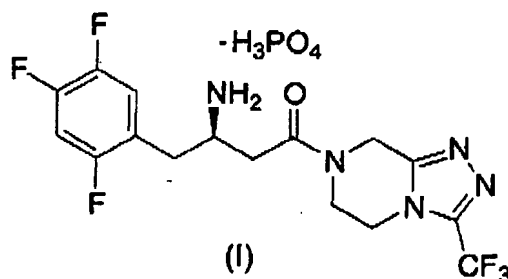
Claim 22 (withdrawn) The crystalline anhydrate Form III of Claim 12 characterized by the differential scanning calorimetric (DSC) curve of FIG. 14.

Serial No.: 10/569,566

Case No.: 21461P

Page No.: 6

Claim 23 (withdrawn) A dihydrogenphosphate salt of (2*R*)-4-oxo-4-[3-(trifluoromethyl)-5,6-dihydro[1,2,4]triazolo[4,3-*a*]pyrazin-7(8*H*)-yl]-1-(2,4,5-trifluorophenyl)butan-2-amine of structural formula I:



characterized as being a crystalline desolvated anhydrate Form II.

Claim 24 (withdrawn) The crystalline desolvated anhydrate Form II of Claim 23 characterized by characteristic reflections obtained from the X-ray powder diffraction pattern at spectral d-spacings of 7.09, 5.27, and 4.30 angstroms.

Claim 25 (withdrawn) The crystalline desolvated anhydrate Form II of Claim 24 further characterized by characteristic reflections obtained from the X-ray powder diffraction pattern at spectral d-spacings of 18.56, 9.43, and 4.19 angstroms.

Claim 26 (withdrawn) The crystalline desolvated anhydrate Form II of Claim 25 further characterized by characteristic reflections obtained from the X-ray powder diffraction pattern at spectral d-spacings of 6.32, 5.82, and 3.69 angstroms.

Claim 27 (withdrawn) The crystalline desolvated anhydrate Form II of Claim 26 further characterized by the X-ray powder diffraction pattern of FIG. 6.

Claim 28 (withdrawn) The crystalline desolvated anhydrate Form II of Claim 23 characterized by a solid-state fluorine-19 MAS nuclear magnetic resonance spectrum showing signals at -65.1, -104.9, and -120.1 p.p.m.

Serial No.: 10/569,566

Case No.: 21461P

Page No.: 7

Claim 29 (withdrawn) The crystalline desolvated anhydrate Form II of Claim 28 further characterized by a solid-state fluorine-19 MAS nuclear magnetic resonance spectrum showing signals at -80.3, -94.5, -134.4, and -143.3 p.p.m.

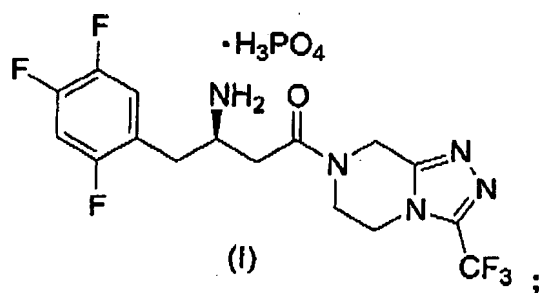
Claim 30 (withdrawn) The crystalline desolvated anhydrate Form II of Claim 29 further characterized by the solid-state fluorine-19 MAS nuclear magnetic resonance spectrum of FIG. 8.

Claim 31 (withdrawn) The crystalline desolvated anhydrate Form II of Claim 23 characterized by the solid-state carbon-13 CPMAS nuclear magnetic resonance spectrum of FIG. 7.

Claim 32 (withdrawn) The crystalline desolvated anhydrate Form II of Claim 23 characterized by the thermogravimetric analysis curve of FIG. 10.

Claim 33 (withdrawn) The crystalline desolvated anhydrate Form II of Claim 23 characterized by the differential scanning calorimetric (DSC) curve of FIG. 9.

Claim 34 (withdrawn) A dihydrogenphosphate salt of (2R)-4-oxo-4-[3-(trifluoromethyl)-5,6-dihydro[1,2,4]triazolo[4,3-a]pyrazin-7(8H)-yl]-1-(2,4,5-trifluorophenyl)butan-2-amine of structural formula I:



characterized as being a crystalline solvate wherein the solvate is selected from the group consisting of acetone solvate, acetonitrile solvate, methanolate, ethanolate, 1-propanolate, and 2-propanolate.

Serial No.: 10/569,566
Case No.: 21461P
Page No.: 8

Claim 35 (withdrawn) The crystalline solvate of Claim 34 wherein said solvate is an ethanolate.

Claim 36 (withdrawn) The crystalline ethanolate of Claim 35 characterized by characteristic reflections obtained from the X-ray powder diffraction pattern at spectral d-spacings of 7.09, 5.27, and 4.30 angstroms.

Claim 37 (withdrawn) The crystalline ethanolate of Claim 36 further characterized by characteristic reflections obtained from the X-ray powder diffraction pattern at spectral d-spacings of 18.56, 9.43, and 4.19 angstroms.

Claim 38 (withdrawn) The crystalline ethanolate of Claim 37 further characterized by characteristic reflections obtained from the X-ray powder diffraction pattern at spectral d-spacings of 6.32, 5.82, and 3.69 angstroms.

Claim 39 (withdrawn) The crystalline ethanolate of Claim 38 further characterized by the X-ray powder diffraction pattern of FIG. 16.

Claim 40 (withdrawn) The crystalline ethanolate of Claim 35 characterized by a solid-state fluorine-19 MAS nuclear magnetic resonance spectrum showing signals at -64.7, -104.5, and -121.9 p.p.m.

Claim 41 (withdrawn) The crystalline ethanolate of Claim 40 further characterized by a solid-state fluorine-19 MAS nuclear magnetic resonance spectrum showing signals at -94.3, -117.7, -131.2, and -142.6 p.p.m.

Claim 42 (withdrawn) The crystalline ethanolate of Claim 41 further characterized by the solid-state fluorine-19 MAS nuclear magnetic resonance spectrum of FIG. 18.

Claim 43 (withdrawn) The crystalline ethanolate of Claim 35 characterized by the solid-state carbon-13 CPMAS nuclear magnetic resonance spectrum of FIG. 17.

Serial No.: 10/569,566

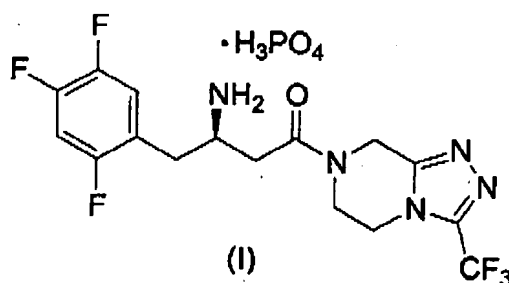
Case No.: 21461P

Page No.: 9

Claim 44 (withdrawn) The crystalline ethanolate of Claim 35 characterized by the thermogravimetric analysis curve of FIG. 20.

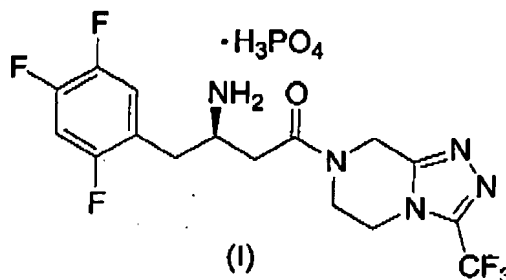
Claim 45 (withdrawn) The crystalline ethanolate of Claim 35 characterized by the differential scanning calorimetric (DSC) curve of FIG. 19.

Claim 46 (withdrawn) A drug substance which is the dihydrogenphosphate salt of (2*R*)-4-oxo-4-[3-(trifluoromethyl)-5,6-dihydro[1,2,4]triazolo[4,3-*a*]pyrazin-7(8*H*)-yl]-1-(2,4,5-trifluorophenyl)butan-2-amine of structural formula I:



comprising a mixture of crystalline anhydrate Form I and crystalline anhydrate Form III.

Claim 47 (withdrawn) A dihydrogenphosphate salt of (2*R*)-4-oxo-4-[3-(trifluoromethyl)-5,6-dihydro[1,2,4]triazolo[4,3-*a*]pyrazin-7(8*H*)-yl]-1-(2,4,5-trifluorophenyl)butan-2-amine of structural formula I:



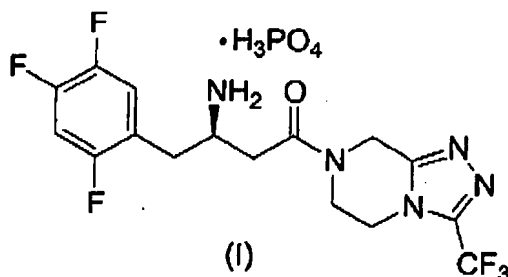
comprising a detectable amount of crystalline anhydrate Form I or crystalline anhydrate Form III or a mixture thereof.

Serial No.: 10/569,566

Case No.: 21461P

Page No.: 10

Claim 48 (withdrawn) A dihydrogenphosphate salt of (2*R*)-4-oxo-4-[3-(trifluoromethyl)-5,6-dihydro[1,2,4]triazolo[4,3-*a*]pyrazin-7(8*H*)-yl]-1-(2,4,5-trifluorophenyl)butan-2-amine of structural formula I:



comprising substantially all by weight of crystalline anhydrate Form I or crystalline anhydrate Form III or a mixture thereof.

Claim 49 (currently amended): A pharmaceutical composition comprising a ~~prophylactically or~~ therapeutically effective amount of the crystalline anhydrate form salt of Claim 1 ~~or Claim 12 or a mixture thereof~~ in association with one or more pharmaceutically acceptable carriers or excipients.

Claim 50 (currently amended): A method of treating Type 2 diabetes comprising administering to a patient in need of such treatment a therapeutically effective amount of the crystalline anhydrate form salt according to Claim 1 ~~or Claim 12 or a mixture thereof~~.

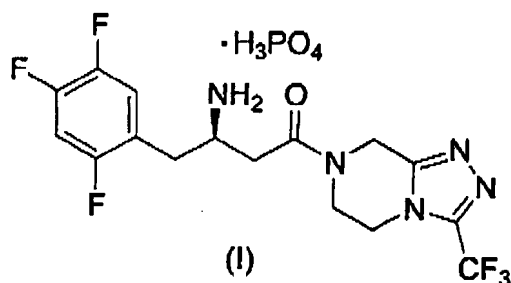
Claims 51-52 (cancelled)

Claim 53 (new): A crystalline anhydrate form of the dihydrogenphosphate salt of (2*R*)-4-oxo-4-[3-(trifluoromethyl)-5,6-dihydro[1,2,4]triazolo[4,3-*a*]pyrazin-7(8*H*)-yl]-1-(2,4,5-trifluorophenyl)butan-2-amine of structural formula I:

Serial No.: 10/569,566

Case No.: 21461P

Page No.: 11



characterized by a Differential Scanning Calorimetry (DSC) melting endotherm with an onset temperature of 215 °C, a peak temperature of 217 °C, and an enthalpy of 221 J/g.

Claim 54 (new): A drug substance comprising a detectable amount of the crystalline anhydrate form of Claim 1.

Claim 55 (new): The drug substance of Claim 54 comprising about 5% to about 100% by weight of said crystalline anhydrate form.

Claim 56 (new): The drug substance of Claim 54 comprising about 10% to about 100% by weight of said crystalline anhydrate form.

Claim 57 (new): The drug substance of Claim 54 comprising about 25% to about 100% by weight of said crystalline anhydrate form.

Claim 58 (new): The drug substance of Claim 54 comprising about 50% to about 100% by weight of said crystalline anhydrate form.

Claim 59 (new): The drug substance of Claim 54 comprising about 75% to about 100% by weight of said crystalline anhydrate form.

Claim 60 (new): The drug substance of Claim 54 comprising substantially all by weight of said crystalline anhydrate form.